U.S. AFOSR DURIP-FY95 Grant

An Intelligent Reactor for Controlled Sputter Deposition of Ceramic Oxide Nanolaminates

Principal Investigator: Carolyn R. Aita
University of Wisconsin-Milwaukee
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Program Manager: Dr. Alexamder Pechenik

FINAL TECHNICAL REPORT

Description of instrumentation and use:

A single piece of equipment was purchased, consisting of a multiple cathode, radio frequency-excited sputter deposition apparatus that is entirely automated. The equipment is custom-made, was designed and built by TM Vaccuum Products, Inc., 630 Warrington Avenue, Cinnaminson, NJ. A contact person is Louis Mozdzen at (609) 829-2000 ext 108. A detailed description of the equipment is attached. The equipment does not differ from that described in the original grant proposal.

The equipment is used to fabricate thin film ceramic nanolaminates. These nanolaminates consist of multilayer stacks in which the consistents are usually polycrystalline. Unique mechanical and chemical properties are achieved because of the nanoscale of each bilayer. These properties include transformation-toughening, enhanced hardness, high temperature stability, and corrosion resistance. A related patent entitled "Multilayer Nanolaminates Containing Polycrystalline Zirconia," has been issued (US No. 5,472,795, C.R. "Atita", 12/5/95, name misspelling correction issued 5/17/96). This patent was developed under DoD funding. To date we have used the equipment to

fabricate multilayers of zirconia-alumina, zirconia-yttria, yttria-alumina, titania-zirconia, and titanium nitride-aluminum nitride.

Ongoing research projects for which the instrumentation is used:

1. C.R. Aita, USARO. "Nanoscale Multialyer Zirconia-Alumina Films for Superior Fracture Toughness." 9/93-6/97, $177,018. (DAAH04-93-G-0238)

2. C.R. Aita, USARO DoD AASERT Grant. "Zirconia-Alumina-ITO Nanolaminates for Transparent, Conducting, Transformation-Toughening Coatings." 6/95-5/98, $104,184. Topic modified to include other nanolaminate combinations. (DAAH04-95-1-0242).

3. C.R. Aita, Shared Research Equipment Program Grant, Oak Ridge National Laboratory. "Mechanical Behavior of Ceramic Nanolaminate Coatings." 10/96-9/97, $4,400 (travel funds)+free equipment use at ORNL.

Publications resulting from the above projects:

1. "Tetragonal zirconia growth by nanolaminate formation." C.M. Scanlan, M.Gajdardziska-Josifovska, and C.R. Aita, Appl. Phys. Lett. 64, 3548 (1994).

2. "The effect of layer thickness on polycrystalline tetragonal ZrO_2 growth in ZrO_2-Al_2O_3 multilayer nanolaminates." C.M. Scanlan, M. Gajdardziska-Josifovska, M.D. Wiggins, and C.R. Aita, Proc. Materials Research Soc. 343, 481 (1994).

3. "Zirconia-alumina nanolaminate coatings: Sputter deposition, phase control, and thermal stability." C.R. Aita, M.D. Wiggins, B. Tian, C.M. Scanlan, and M. Gajdardziska-Josifovska, in Elevated Temperature Coatings: Science and Technology I (edited by N.B. Dahotre et al., TMS, Warrendale, PA, 1995) pp. 252-259. Invited paper at 1994 TMS Fall Meeting.

4. "Sputter deposited nanostructured ceramic coatings for corrosion wear and resistance." C.R. Aita, in Advances in Coatings Technology for Corrosion and Wear Control Coatings (edited by A.R. Srivatsa et al., TMS, Warrendale, PA, 1995) pp. 235-254. Invited paper at 1995 TMS Annual Meeting.

5. "The transformation structure of zirconia-alumina nanolaminates studied by high resolution electron microscopy." M. Gajdardziska-Josifovska and C.R. Aita, J. Appl. Phys. 79, 1315 (1996).

6. "Thermodynamics of tetragonal zirconia formation in a nanolaminate film." C.R. Aita, M.D. Wiggins, R.Whig, C.M. Scanlan, and M. Gajdardziska-Josifovska, J. Appl. Phys. 79, 1176 (1996).

7 "Zirconia-alumina transformation-toughening nanolaminate coatings." C.R. Aita, M. Gajdardziska-Josifovska, and M.D. Wiggins, in Processing and Fabrication of Advanced Materials IV (edited by T.S. Srivatsan and J.J. Moore, TMS, Warrendale, PA, 1996) pp. 237-249. Invited paper at 1995 TMS Fall Meeting.

8. " Phase formation in sputter deposited titania." M.D. Wiggins, M.C. Nelson, and C.R. Aita, J. Vac. Sci. Technol. A 14, 772 (1996).

9. "Crystallization kinetics of rutile formation from amorphous titania films." M.D. Wiggins, M.C. Nelson, and C.R. Aita, Proc. Mater. Res. Soc. 398, 381 (1996).

10. "High resolution electron microscopy of sputter-deposited zirconia-alumina nanolamiantes." M.A. Schofield, R. Whig, C.R. Aita, and M. Gajdardziska-Josifovska, Proc. Mater. Res. Soc. 403, 297 (1996).

11. "Sputter deposition of boron nitride using neon-nitrogen discharges." R.B. Heil and C.R. Aita, J. Vac. Sci. Technol. A 15 (1997) in press.

12. "Nanostructured ceramic coatings: Zirconia single layers and nanolaminates." C.R. Aita, Processing and Fabrication of Advanced Materials V (edited by T.S. Srivatsan and J.J. Moore, TMS, Warrendale, PA, 1996) in press. Invited paper at 1996 TMS Fall Meeting.

13. "Electron-beam induced transformations in zirconia-alumina nanolaminates: an *in situ* high resolution electron microscopy study." M.A. Schofield, M. Gajdardziska-Josifovska, R. Whig, and C.R. Aita, Proc. 54nd Ann. Mtg. Mircoscopy Society of America (edited by G.W. Bailey et al. (San Francisco Press, San Francisco, CA, 1996) pp. 690-691.

Invited papers at national/international symposia resulting from the above projects:

1. "Sputter deposited zirconia-alumina nanolaminates: fabrication, phase control, and thermal stability." C.R. Aita The Minerals, Metals, & Materials Society (TMS) Fall Meeting, Rosemont, IL, 1994.

2. "Nanostructured ceramic and multilayer nanolaminate coatings for corrosion and wear resistance. C.R. Aita The Minerals, Metals, & Materials Society (TMS) Annual Meeting, Las Vegas, NV, 1995.

3. "Transformation-toughening ceramic nanolaminate coatings." C.R. Aita The Minerals, Metals, & Materials Society (TMS) Fall Meeting, Cleveland, OH, 1995

4. "Nanostructured ceramic coatings" C.R. Aita The Minerals, Metals, & Materials Society (TMS) Fall Meeting, Cincinnati, OH, 1996.

5. "Reactive physical vapor deposition processes." C.R. Aita,.International Materials Research Congress, Cancun, Mexico, 1996.

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F.O.B.	TERMS	DELIVERY	REFERENCE	Inquiry No.	BULLETIN NO.

QUANTITY	UNIT	ITEM DESCRIPTION	Commodity Code	UNIT PRICE	TOTAL
1	System	Automated Sputter Deposition System designed to deposit multi-layer stacks of alternating materials via RF diode sputtering specifically for transformation-toughening Oxide Nano-laminates [SPECIFICATIONS DETAILED ON ATTACHED 5 pp.]	For Purchasing Office Use Only		$259,968.00

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Approvals:

Department Chairperson George S Baker Date 23 Aug 95

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SPUTTER DEPOSITION SYSTEM
FOR TRANSFORMATION - TOUGHENING
OXIDE NANOLAMINATES

VACUUM CHAMBER

The vacuum vessel is constructed from 304 stainless steel. Chamber diameter is 24" and the chamber measures 14" tall. The chamber base plate contains the following penetrations:

- One (1) 6" pumping port which mates with cryopump.
- One (1) 1½" diameter roughing port.
- One (1) 1" compression fitting for hot filament ion gauge tube.
- One (1) ¼ pipe tap for thermocouple gauge tube.
- One (1) Central penetration for substrate table.
- One (1) penetration for admission of process gas.
- One (1) penetration for admission of venting gas.

The top of the chamber is fitted with a 24" diameter sealing flange. This main sealing flange seals the chamber to the removable lid via a Viton O-ring. The chamber side wall contains the following penetrations:

- One (1) shuttered 4" viewport located at the front of the chamber.
- One (1) 4" quartz window to accommodate customer supplied optical emission spectrometer.
- Two (2) 2-3/4 conflat flanges for crystal monitor heads.

The chamber top plate is removable and contains the following penetrations:

- Four (4) 5" diameter holes, two (2) are fitted with sputtering cathodes, one (1) is fitted with a radiant heater and the final hole is blanked for future use.

VACUUM PUMPING

The system is rough pumped by a 20 cfm two stage mechanical pump. High vacuum pumping is provided by a 4000 liter/second (for water) cryopump with water cooled liquid helium compressor.

VACUUM VALVING

All vacuum valves are electropneumatically operated and all vent and gas admission valves are solenoid operated. High vacuum valving is provided by a 6" A.S.A. stainless steel gate valve. A 1½" right angle stainless valve provides rough pump valving. A 1" right angle valve provides for pump-out of the cryopump after regeneration. A ½" solenoid valve provides purge gas admission for use during the cryo regeneration process.

VACUUM GAUGING

The dual function vacuum gauge provides both rough vacuum and high vacuum readouts. The rough vacuum portion of the gauge provides readouts for two (2) thermocouple gauge tubes via a dual bar graph display. Each gauge tube has two (2) adjustable set points.

The high vacuum portion of the gauge reads out via a digital display that reads from 10^{-4} *TORR* down to 10^{-8} *TORR*. The high vacuum readouts are taken from a hot filament ion gauge tube.

VACUUM PIPING

The vacuum pumping manifold is constructed from Type K hard copper tubing and is silver soldered into a leak tight rigid assembly.

SYSTEM FRAME

The vacuum chamber and pumps are mounted within a structural steel frame which is covered by removable steel skins.

SUBSTRATE TABLE

A 22" diameter rotating aluminum substrate table is centrally located in the bottom of the chamber. The table rotates via external drive motor between 0-30 rpm. The table is electrically isolated from the chamber and is mounted on a FerroFluidic feedthrough.

SPUTTER SOURCES

Two (2) 5" diameter RF diode cathodes are mounted on the chamber top plate. The power and water connections to the cathodes are external to the sputtering system. Each cathode is fitted with a pneumatically operated shutter, which is primarily used as a deposition catcher during pre-sputtering.
(Cathode cleanup.)

SUBSTRATE HEAT

A 2 KW radiant heater provides for heating of the substrates when necessary. Heating can be accomplished while the substrate table is stationary or rotating. The heater is mounted on the chamber top plate and electrical connections are external to the vacuum.

SPUTTER POWER

Two (2) dedicated RF power supplies (13.56 Mhz) provide sputter power. Each RF supply is rated at 2 KW. Two (2) auto tune networks are provided for the power supplies. Additionally a phase shifter is included. The cathodes can be operated either simultaneously or sequentially without causing RF interference.

PROCESS GAS SYSTEM

A two (2) gas flow control and pressure control system is comprised of the following components:

- Two (2) thermal mass flow controllers, 1 each for argon and oxygen. (please specify flow range).
- One (1) four channel, four gas flow controller electronics unit.
- One (1) high accuracy capacitance manometer (0-100 millitorr range).
- One (1) capacitance manometer electronic control unit.
- One (1) servo controlled, 6" diameter low vane throttle valve.
- One (1) exhaust control valve electronics unit.

DEPOSITION THICKNESS MONITORS

- Two (2) water cooled crystal thickness monitors (one beneath each cathode) are located within the vacuum chamber.
- A single electronics control unit services both crystal monitor heads.
- One or both cathodes can be powered at a given time. Each crystal monitor can be programmed to shut down sputter power from the cathode it is monitoring when the required thickness is reached.

CHAMBER HOIST

The flat chamber lid containing two cathodes, radiant heater and blanked off fourth cathode port is lifted by an air/oil hoist. The lid can travel up to 18" vertically from its sealed position on the chamber. The hoist is operated by a momentary raise/lower switch.

SYSTEM OPERATIONAL MODES

AUTOMATIC MODE

In this mode the operator would load the system with one (1) or more substrates, lower the chamber lid and push the "auto pump" button.

The system will automatically pump from atmosphere to high vacuum. When high vacuum is reached, the operator will initiate the automatic coating cycle by pushing the "process start" button. The system will automatically deposit alternate layers of two (2) reactively sputtered materials. Sputter parameters have been previously established and set. The system will automatically rotate the substrate table. At the end of a predetermined number of deposited layers the system will stop the coating process.

The operator pushes the "system vent" button and the vacuum chamber vents to atmosphere.

MANUAL MODE

In this operational mode the system vacuum valves are actuated by manually pushing lighted valve buttons on the control panel. The deposition portion of the cycle remains the same as described above. In this mode all safety interlocks are active. The operator cannot improperly sequence the vacuum valves.

MAINTENANCE MODE

In this mode, all functions are carried out manually with interlocks disabled.

CAUTION: This is a key locked mode and should only be used by trained maintenance personnel who are intimately familiar with the operation of the system. This mode is usually employed when diagnostic and/or repair procedures are conducted.

AUTO REGEN MODE

The system is equipped with an automatic cryopump regeneration mode. By switching the system to this mode and pushing the AUTO REGEN button, the system will automatically go through a complete cryopump regeneration cycle and return itself to a "READY TO RUN" condition. The AUTO REGEN cycle usually takes a total of about 5-6 hours.

SYSTEM INTERLOCKS

This system is equipped with the following safety interlocks:

- Vacuum interlock: Monitors sequencing of vacuum valves so that valves cannot be opened unless proper pressure conditions exist. Prevents hoist from operating when chamber is under vacuum.
- Electrical interlock: Prevents sputter power from being applied unless chamber is under vacuum.
- Water interlock: Sputtering power and cryo compressor cannot be operated unless water flow is present.
- Compressed air interlock: Vacuum valves and hoist cannot be operated unless compressed air pressure is present.
- Cabinet interlock: High voltage in electrical system will not operate unless all cabinet doors are closed.

UTILITIES REQUIRED

- 208 volt, 3 phase, 60 hz, 100 amp electrical service.
- Compressed air - 2 cfm @ 90-120 psig.
- Water. 3 gpm at 55° Fahrenheit.
- Process gas: Argon, dry nitrogen, and oxygen from bottled sources. Regulated down to 10-15 psig.

  The customer will provide an optical emission spectrometer to be used with the system, and two (2) 5" sputtering targets.

WARRANTY

The system is warranted for one (1) year from date of shipment. Warranty includes parts and labor but does not include consumables such as sputtering targets, pump fluids, and O-rings.

The system is available for $259,968.00 F.O.B., Cinnaminson, NJ

Delivery will be via padded electronics van in 18-20 weeks after receipt of order.

Payment terms are:

- 20% with purchase order.
- 20% upon approval of engineering drawings.
- 20% upon commencement of construction.
- 20% upon acceptance at T-M Vacuum.
- 20% net 30 upon shipment.

John Strada

T-M VACUUM PRODUCTS, INC.

John Strada
Sales Manager

JS/cam